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Influence of mould thermal properties on the replication of micro parts via injection moulding

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Abstract

The surface quality that results when replicating micro features is one of the most important process characteristics in micro injection moulding, and it constitutes a manufacturing constraint in applying the technique to a wider range of micro engineering applications. Moulding micro features with a high aspect ratio is a critical task, in particular when the feature width is small, due to a faster temperature decrease than in macro/meso scale cavities. In order to investigate the influence of the thermal diffusivity of the mould material in micro structured surfaces replication, in this paper two moulds, made respectively of tool steel and zirconia ceramic composite, have been used to replicate a micro structured surface. Micro Electrical Discharge Machining (μEDM) was employed to manufacture both the steel and the ceramic mould. The thermal diffusivity of the mould materials was measured in order to relate it to the degree of replication. Then, micro features were replicated via micro injection moulding, at the same controlled process conditions, and the replication degree was measured by means of an optical coordinate measurement machine. The results of the experimental tests display a sharp improvement of the quality of the micro structure replicated with the ceramic mould, that is when using a mould material with low thermal diffusivity. This effect, which is related to the ability of the material to delay the polymer skin solidification when the cavity is filled in, can be effectively exploit to enhance the capabilities of the current micro injection moulding technologies in manufacturing components with features characterized by higher complexity and aspect ratio.

Keywords: micro injection moulding; surface replication; micro electrical discharge machining

1. Introduction

The surface quality that results when replicating micro features is one of the most important process characteristics in micro injection moulding, and it constitutes a manufacturing constraint in applying the technique to a wider range of micro engineering applications. To achieve the required accuracy and prevent premature material freezing when producing high-aspect-ratio micro features, high injection pressures and mould temperatures are usually required [1]. Moulding micro features with a high aspect ratio (depth vs. width of feature) is a critical task, especially when the feature width is small due to a faster temperature decrease than in regular-sized cavities. A polymer is frozen when its temperature drops below the no-flow or solidification temperature. While the polymer is filling a micro channel, the thin, frozen layers grow rapidly from the channel walls due to the very fast heat loss. The polymer stops flowing when the frozen layer covers the whole cross section of the micro channel. To overcome this difficulty, high injection speed and mould temperature would reduce heat loss from the polymer melt. However, the practical processing conditions are limited by the physical capabilities of the moulding tools [2].

In order to investigate the importance of processing conditions on micro features filling, several research groups have conducted injection moulding experiments on micro featured parts with well-defined simple geometries. In particular, Despa et al. moulded micro structures with an aspect ratio of 8 and lateral dimensions

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of 90 μm [3]. For the moulding material (high density polyethylene), they found that a mould temperature of 135°C allowed the complete filling of the micro voids. Yao and Kim achieved the complete replication of 40 μm wide micro channels with an aspect ratio of 10 at a mould temperature of 140°C [4]. On the contrary, the replication degree was close to zero attemperatures lower than 90°C. Winberger-Friedl studied the effect of the mould temperature on the filling depth of polycarbonate microstructures, 0.2 μm wide and 0.8 μm deep, on grating optical elements. The depth of filling varied from 30 to 100% within a temperature range of 7°C. Only at a mould temperature of 152°C, which is above the glass transition of polycarbonate, the structures were filled completely [5]. Yu et al. conducted injection moulding experiments while filling with unidirectional flow a rectangular cavity presenting micro features 20 times thinner than the main cavity [6]. They observed that the filling process consists essentially of two stages. The first one is characterized by the competition between the main flow in the base plate and the branch flow in the micro feature. The pressure required to drive the former is much lower than in the micro feature. During the base plate filling, the cavity pressure may not build up to a level high enough to fill in the micro feature and consequently the flow hesitates and cools down. Once the base plate is filled, the second stage starts, which is characterized by a quick raise of the cavity pressure. This pressure would have been high enough to completely fill the micro feature if only the flow had not hesitated freezing at the micro feature entrance. The smaller the feature size, the more likely this happens. This trend is further confirmed by Wimberger-Friedl's result, in which the micro feature size is in the submicron range. The flow in a 0.2 μm channel seems to depend solely on the mould temperature. A mould temperature above the polymer no-flow temperature is essential in this case.

From an extensive scientific literature review, it clearly emerges that almost all the attempts carried out so far to improve the replication of micro structured surfaces have been mainly focused on the process parameters (e.g. best combination of mould temperature, injection speed and melt temperature). On the other hand, little attention has been paid to the mould material properties. On the other hand, to achieve a high degree of replication of the finished parts, mould materials with low thermal diffusivity should be desirable.

This aspect is here considered, and in order to investigate the influence of mould diffusivity on micro structured surfaces replication, in this paper two moulds, made respectively of tool steel and zirconia ceramic composite, are used to replicate moulded micro features with variable size. Zirconia ceramics are indeed excellent thermal insulators; besides they are the toughest ceramics.

2. Experimental

2.1. Materials

For the experimental work, a commercial polystyrene (PS) resin (Edistir RC 600 by Polimeri Europa) was used. It is a special medium impact polystyrene grade exhibiting very high gloss; it is designed to be used in injection moulding where an excellent surface finish is required. Polystyrene is relevant in micro injection moulding for its very high flow ability, good biocompatibility, high optical clarity, high transparency and high impact strength compared to silicon or glass. The polymer has a solid density of 1.04 g/cm^3. The melt index (MI) is 6.00 g/10 min (200°C, 5.00 kg). The glass transition temperature is 95°C.

In this study, two mould inserts were used, made respectively of a premium grade stainless tool steel (Stavax® ESR by Bohler-Uddeholm) and a ZrO_2-TiN ceramic composite (KGS-40 grade, commercially available by NTK Technical Ceramics). The zirconia ceramic was specifically selected because of the superior thermal insulating and mechanical properties provided. The material offers very low thermal diffusivity, improved fracture toughness and exceptional high flexural strength, up to 1700 MPa. According to an in–house experimental campaign, this value, however, may degrade up to about 1200 MPa, depending on the particular procedure used for the sample preparation (in the specific case, thermal treatment plus grinding), and the degree of surface finishing achieved (Ra 0.25 μm and Rt 1.86 μm); thus, confirming the strong influence that manufacturing and presence of defects have on the final performance of ceramics.

Table 1. Material properties of the ZrO_2-TiN ceramic composite used as mould material (data provided by the material manufacturer).

Density (g/cm^3)	5.8
Vickers Hardness Kg/mm^2 (HV_{10})	1350
Fracture Toughness (MPa m$^{1/2}$)	9.7
Flexural Strength (MPa)	1700 @ 3pt
	(1168 ± 81 @ 3pt*)
Young's Modulus (GPa)	280
Thermal Expansion Coefficient (1/K)	10*10^{-6}
Thermal conductivity (W/mK^{-1})	8
Electrical resistivity (Ωcm)	1.5x10^{-3}

* in-house value

Additionally, the addition of the electrically conductive secondary phase (TiN) within the ZrO_2 matrix allow the machining of this material by Electrical Discharge Machining (EDM); thus, overcoming to the overall difficulties of machining ceramics into the final states, especially as 3D complex shapes or small features are required [7]. Table 1 lists the material properties of the ZrO_2-TiN ceramic composite; instead the thermo-physical properties, including thermal conductivity, specific heat and thermal diffusivity, of both insert materials, are reported in Table 2 and 3. The properties were measured at Petroceramic S.p.A. by means of a laser flash system, the Netzsch LFA 457 MicroFlash, according to the ASTM E-1461 standard.

Table 2. Thermo-physical properties of the stainless steel Stavax® ESR by Bohler-Uddeholm, used for the mould insert (standard deviations in brackets)

Temperature [°C]	Specific Heat [J/g*K]	Thermal Conductivity [W/m*K]	Thermal Diffusivity [mm^2/s]
25	0.42 (0.08)	12.61 (0.13)	3.79 (0.04)
200	0.44 (0.08)	14.91 (0.02)	4.32 (0.01)

Table 3. Thermo-physical properties of the ZrO_2-TiN KGS-40 by NTK Technical Ceramics, used for the mould insert (standard deviations in brackets)

Temperature [°C]	Specific Heat [J/g*K]	Thermal Conductivity [W/m*K]	Thermal Diffusivity [mm^2/s]
25	0.52 (0.05)	8.44 (0.05)	2.82 (0.02)
400	0.63 (0.07)	7.13 (0.05)	1.97 (0.01)
800	0.67 (0.08)	6.56 (0.03)	1.72 (0.01)

2.2. Mould manufacturing

The mould cavity is hosted in a square insert of 15×15 mm^2 with a thickness of 2.5 mm. Specifically, the ceramic mould insert was firstly cut via wire-EDM on a Robofil 2000 (Charmilles technology). The alignment and mould surfaces were all cut in one single operation in order to assure tight tolerance in planarity, and finished by selecting a WC fine cut technology. The pulse frequency parameter was specifically modified to avoid continuously breaking of the wire during machining, and to assure smooth wire cutting. The mould surface was successively machined via micro-EDM milling for the realisation of the mould features.
Specifically, the machine used was a Sarix SX-200, equipped with a micro fine pulse generator, a wire dress unit for micro tool electrode fabrication and a laser scan micrometer for micro tool diameter control. Tungsten carbide solid rods were used as tool electrodes and mould manufacturing was carried out in a low viscosity hydrocarbon oil as dielectric medium (HEDMA 111). The micro EDM milling process parameters were selected based on previous experience, in the case of the ZrO_2-TiN [7,8], and among the technology tables provided by the machine constructor, as far as the tool steel was concerned. Based on an in-house experimental campaign of three-point bending tests conducted on EDM ZrO_2-TiN samples machined at similar conditions, a further reduction (~15 %) of the flexural strength of the ceramic material during service due to micro-EDM machining is also expected (reference in-house value as reported in Table 1).

Additionally, specific self-learning micro-milling routines on sacrificial workpieces, made of the same material of the mould inserts, were performed in order to refine the value of the adjustment factor peculiar for compensating the tool wear along the tool axis. Finally, the tool paths for milling operations were programmed using a dedicated CAM software.

A circular micro filter was selected as reference model for the experimental campaign. Figure 1 shows the model of the cavity. The outer diameter is 1.95 mm. The inner ribs have a square section of 0.15 x 0.15 mm. The spacing between them measures 50 µm.

Fig. 1. Model of the reference part used for the experimental campaign.

2.3. Injection moulding experiments

The injection moulding experiments were conducted on a Wittmann-Battenfeld MicroPower 15 all-electric micro-injection moulding machine. The mould cavity considered in this study is placed at the end of a trapezoidal cold runner 27.5 mm long, 3 to 2 mm wide and 1.5 mm thick, connected through a rectangular gate 0.4 mm long, 0.5 mm wide and 0.15 mm thick. The polymer was injected at a constant flow rate of 3.93 cm^3/s, limiting the maximum injection pressure (switch-over) at 220 MPa. The packing pressure was maintained at a constant value of 150 MPa for 5 s. These process settings were especially selected to yield incomplete filling and to guarantee the highest degree of precision in order to isolate the effect of mould thermal diffusivity, mould temperature and their interaction on the replication degree. The moulded part was further cooled in the mould for 15 s and then ejected. The melt temperature in the feeding zone was maintained at 220°C. For each of the two mould materials, the mould temperature was varied on three levels, namely 40, 60 and 80°C, according to a general full factorial plan.

During injection, an automatic execution of the process (including part ejection and handling) was performed for each treatment. Firstly, 50 cycles where carried out to stabilize the process. Subsequently, 10 parts obtained from the following 30 cycles have been randomly collected and analysed.

2.4. Characterization of the moulded parts

Optical microscopy was employed to picture the obtained degree of replication. A precise characterisation of the microscopic structures was performed by means of a multi-sensor coordinate measurement machine (Video Check IP 400, Werth Messtechnik, Giessen, Germany). Each structure was measured with the same instrument setting in order to allow a straightforward comparison of the results. The collected samples were measured at the 7 different positions, corresponding to the centres of the ribs. The obtained micro features were quite regular, showing an even filling pattern except for the extremities, which are clearly affected by the filling resistance of the side walls. The replication degree was eventually calculated by lumping together the filling length of all the ribs. Representative sample parts for each of the 6 treatments are shown in Figure 2.

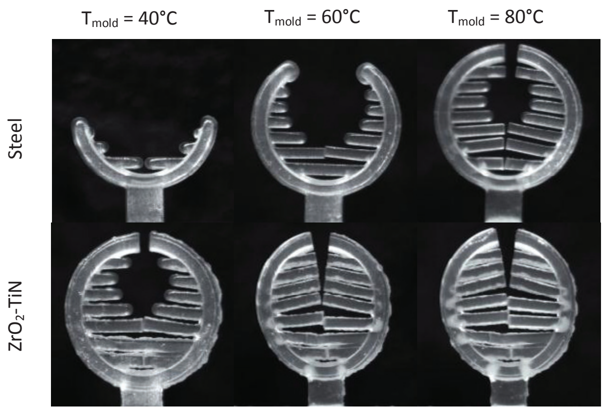

Fig.2 Sample parts collected for each of the 6 treatments.

3. Experimental results and discussion

Filling length results are reported in Table 4 according to thermal diffusivity of the mould material. A statistical analysis of the filling results was performed and the effects of mould thermal diffusivity and mould temperature calculated. As can be seen in Figure 3, both factors have a large effect on the filling percentage. In particular, it can be clearly observed that the degree of replication is strongly increased by employing a mould material having a lower thermal diffusivity, and the filling fraction values are significant higher when using zirconia as mould material, especially at low injection temperature.

Table 4. Average filling length results. Values are expressed in mm (standard deviations in brackets) and as percentage of filling.

Mold thermal diffusivity [mm^2/s]	Mold Temperature [°C]		
	40	60	80
3.96	0.51 (0.11)	2.15 (0.64)	4.45 (0.28)
	11%	47%	97%
2.69	3.42 (0.27)	4.58 (0.00)	4.58 (0.00)
	75%	100%	100%

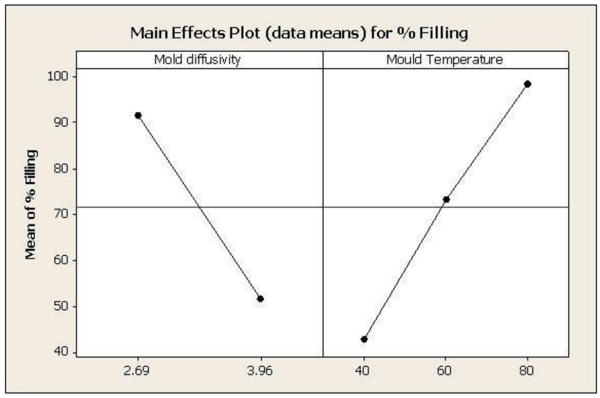

Fig. 3. Main effect plot for replication degree as function of mould thermal diffusivity and mould temperature.

On the other hand, the ceramic insert display severe damages after use and significant wear, especially at the top edges of the filter ribs (Figure 4). The result is retained to be a consequence of the lower mechanical strength of the ceramics as compared to the tool steel, and generated during the ejection phase.

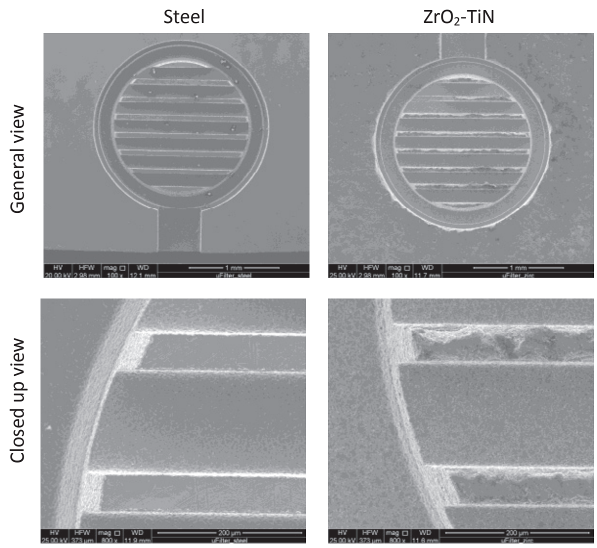

Fig. 4. SEM pictures of the tool steel and ceramic insert after use along with the respective closed up views

A significant reduction of the mechanical performance of the ceramic insert might have been also induced by the manufacturing procedure specifically adopted. As reported in Section 2, the ceramic insert was firstly cut, and surfaces were finished via wire-EDM by using a fine cut, but no dedicated, technology. The actual result is a rough surface, displaying several large craters and defects, which may have been the initiation of severe fracture during service, especially at the edges of the mould rib features, whereupon stress intensification is experienced. The result is a further demonstration of the importance that the manufacturing process has on the performance of ceramics during service.

4. Conclusions

In order to investigate the influence of the thermal diffusivity of the mould material in micro structured surfaces replication, in this paper two moulds, made respectively of tool steel and zirconia ceramic composite, have been used to replicate a micro structured surface. Micro Electrical Discharge Machining (μEDM) was employed to manufacture both the steel and the ceramic mould. The thermal diffusivity of the mould materials was measured in order to relate it to the degree of replication. Then, micro features were replicated via micro injection moulding, at the same controlled process conditions, and the replication degree was measured by means of an optical coordinate measurement machine. The results of the experimental tests display a sharp improvement of the quality of the micro structure replicated with the ceramic mould, that is when using a mould material with low thermal diffusivity. This effect, which is related to the ceramic ability to delay the polymer skin solidification when the cavity is filled in, could be effectively exploit to enhance the capabilities of the actual micro technologies in manufacturing components with feature characterized by higher complexity and aspect ratio. On the other hand, the manufacturing and handling of ceramic moulds requires particular care in order to avoid premature tool wear and damages. This aspect is currently under deep investigation and will receive particular attention in future works.

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